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Cement & Concrete Composites 21 (1999) 293–300

Cement & Concrete Composites

Comparative study of the initial surface absorption and chloride diffusion of high performance zeolite, silica fume and PFA concretes

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Received 9 October 1997; accepted 16 February 1999

Abstract

Zeolite, a type of natural pozzolanic material, has been used in producing blended cement and concrete in China. The purpose of this study is to evaluate the effectiveness of zeolite in enhancing the performance of concrete in comparison with silica fume and pulverized fuel ash (PFA). In the first series of experiments, zeolite, silica fume, and PFA were all used to replace 5%, 10%, 15% and 30% of cement by weight in concrete with water to total cementitious material ratio ($W/(C + P)$) kept at 0.28. The results showed that zeolite decreased bleeding and increased marginally the viscosity of concrete without significantly compromising the slump. And at 15% replacement level, it resulted in 14% increase in concrete strength at 28-day compared with the control concrete. The test results also showed that there existed an optimum replacement level for zeolite to effect a decrease in initial surface absorption and in chloride diffusion of concrete. The test results of the second series of experiments where zeolite, silica fume and PFA were in turn used to replace 10% of cement in concretes with $W/(C + P)$ in the range of 0.27 to 0.45 appeared that zeolite performed better than PFA but was inferior to silica fume in terms of increasing strength, decreasing initial surface absorption and chloride diffusion. It was further found that when $W/(C + P)$ was greater than 0.45, the strength of the concretes incorporating zeolite or PFA (by direct replacement) was lower than that of the control concrete. The micro-structural study on concrete with zeolite revealed that the soluble SiO_2 and Al_2O_3 could react with $\text{Ca}(\text{OH})_2$ to produce C-S-H which densified the concrete matrix. Pozzolanic effect of zeolite improved the microstructure of hardened cement paste and reduced the content of the harmful large pores, hence made concrete more impervious. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: High performance concrete; Zeolite; Silica fume; PFA; Initial surface absorption; Chloride diffusion

1. Introduction

Natural zeolite which is a popular natural pozzolanic material in China is widely and abundantly deposited in the country. It can be easily quarried and processed. It was reported that out of the annual output of about 200 million tonnes of cement in China, more than 20% of the cement was blended with zeolite [1]. Zeolite is also widely used as a cement replacement material in producing concrete structural elements. High performance concrete with high strength (over 80 MPa) and high flowing ability (slump 100–200 mm) was produced with zeolite at water to total cementitious materials ratio $W/(C + P) = 0.28\text{--}0.30$. [2] Treated zeolite can also be used to produce high performance lightweight cellular

concrete [3]. Zeolite also effectively prevented the expansion of concrete due to alkali silica reaction, and was capable of controlling slump loss of high strength concrete when it was used as a carrier for superplasticizer and plasticizer [4,5].

This study evaluates the effectiveness of zeolite in making high performance concrete in comparison with PFA and silica fume concretes. The performances of concrete were assessed in terms of slump of fresh concrete, 3 7-day and 28-day strengths, 10–60 min initial surface absorption, and chloride diffusion. There are two series of experiments in this research. In the first series, the effects of zeolite, silica fume and PFA, which in turn replaced 10%, 15%, and 30% of cement by weight, on the performances of concrete were compared. In the second series, the effects of varying water to total cementitious materials ratios on the performance of control concrete [6], concretes incorporating zeolite, silica fume and PFA were investigated. Finally the effect

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of zeolite on the improvement of microstructure of hardened cement paste was studied.

2. Materials

Binders

(a) Cement: Ordinary Portland cement (OPC) complying with BS4550:Part 2.

(b) Zeolite: Quarried in Du Shi Kou, Hebei Province and ground with a vibration grinder to obtain high specific surface.

(c) PFA: produced in Hong Kong, complying with BS3892:Part 1:1982.

(d) Silica fume: produced and supplied by Master Builders Technologies (Hong Kong) Ltd.

The chemical compositions and physical properties of the ordinary Portland cement, zeolite, silica fume and PFA used in the experiments are summarized in Table 1.

Coarse aggregate: single sized 10 mm and 20 mm crushed granite.

Fine aggregate: river sand in grading zone F in accordance with BS882.

Superplasticizer: naphthalene based with a specific gravity of 1.2 and a solid content of 40%. For the purpose of producing high strength and high flowing concrete, superplasticizer was added to all concrete mixtures in the series 1 concretes and to most of the mixtures in the series 2 concretes.

Table 1
Chemical compositions and physical properties of ordinary Portland cement (OPC), zeolite, PFA and silica fume

Properties	Binder			
	OPC	Zeolite	PFA	Silica fume
Oxide composition				
% by mass				
CaO	64.4	3.67	5.69	0.3
SiO ₂	20.7	65.01	44.92	94
Al ₂ O ₃	5.4	11.85	35.39	0.3
Fe ₂ O ₃	2.3	1.03	4.89	0.8
MgO	0.9	0.48	1.24	0.4
Na ₂ O	0.13	1.65	1.0	1.0
K ₂ O	0.4	1.75	1.0	
SO ₃	2.4	0.02	0.71	0.2
TiO ₂	—	0.14	—	—
Insoluble	0.3	—	3.32	—
Chloride content	<0.05	—	<0.05	—
Loss on ignition	0.97	1.70	5.61	2.8
Specific gravity	3.1	2.16	2.1	2.2
Specific surface area (m ² /kg)	355	750	528	2 × 10 ⁴
Compressive strength (MPa)				
3 day	22.7	—	—	—
7 day	40.7	—	—	—
28 day	58.9	—	—	—

3. Mixture proportions

(1) *Series 1 experiments.* The purposes of the series 1 experiments were to evaluate the effects of zeolite, PFA and silica fume on slump, 7-day, 28-day strength, 10–60 min initial surface absorption, and chloride diffusion of high strength concrete (28-day strength around 100 MPa). The water to total cementitious materials ratio $W/(C + P)$ was kept at 0.28 for this series of study. Superplasticizer was added to attain a slump of about 200 mm. Zeolite, PFA and silica fume were in turn used to replace 5%, 10%, 15% and 30% by weight of cement. The water content was maintained at 154 kg/m³. The fine aggregate content was kept at 527 kg/m³ while the 20 mm and 10 mm single sized coarse aggregates were 819 kg/m³ and 410 kg/m³, respectively. The mixture proportions are given in Table 2.

(2) *Series 2 experiments.* The purposes of the series 2 experiments were to evaluate the effects of zeolite, PFA and silica fume on the performance of concretes with varying $W/(C + P)$ ratios. In this series, the replacement level of cement by zeolite, PFA and silica fume was kept at 10% by weight of cement. The $W/(C + P)$ ratios for the concretes were varied as 0.27, 0.33, 0.39 and 0.45. The mixture proportions are given in Table 3.

4. Specimen preparation, curing and testing

(1) *Mixing procedure.* Prior to mixing the concrete, zeolite was oven-dried at 105°C until constant weight because it could absorb moisture owing to its high water absorption capacity due to its large surface area and large number of micro-passages and cavities in the zeolite crystal structure. The dry materials of concrete

Table 2
Binder content of concrete mixtures used in series 1 experiment ($W/(C + P) = 0.28$)

Concrete code	Cement replacement material and level (%)	Content of cement replacement material (kg/m ³)	Cement (kg/m ³)	Super plasticizer (L/m ³)
Control	—	—	550	8
ZE5	Zeolite, 5	27.5	522.5	8
ZE10	Zeolite, 10	55	495	8
ZE15	Zeolite, 15	82.5	467.5	8
ZE30	Zeolite, 30	165	385	9.5
FA5	PFA, 5	27.5	522.5	8
FA10	PFA, 10	55	495	8
FA15	PFA, 15	82.5	467.5	8
FA30	PFA, 30	165	385	9.5
SF5	Silica fume, 5	27.5	522.5	8
SF10	Silica fume, 10	55	495	8
SF15	Silica fume, 15	82.5	467.5	8
SF30	Silica fume, 30	165	385	13.4

Table 3
Mix proportions for concretes in series 2 experiments

No.	W/(C + P)	Cement replacement material and level (%)	Content of cement replacement material (kg/m ³)	Cement (kg/m ³)	Water (kg/m ³)	20 mm aggregate (kg/m ³)	10 mm aggregate (kg/m ³)	Sand (kg/m ³)	Super-plasticizer (l/m ³)
1	0.27	—	—	500	135	426	851	548	7.3
2	0.33	—	—	500	165	419	837	539	4.7
3	0.39	—	—	500	195	410	819	527	2.7
4	0.45	—	—	500	225	403	805	518	0
5	0.27	Zeolite, 10	50	450	135	426	851	548	7.3
6	0.33	Zeolite, 10	50	450	165	419	837	539	4.7
7	0.39	Zeolite, 10	50	450	195	410	819	527	2.7
8	0.45	Zeolite, 10	50	450	225	403	805	518	0
9	0.27	PFA, 10	50	450	135	426	851	548	7.3
10	0.33	PFA, 10	50	450	165	419	837	539	4.7
11	0.39	PFA, 10	50	450	195	410	819	527	2.7
12	0.45	PFA, 10	50	450	225	403	805	518	0
13	0.27	Silica fume, 10	50	450	135	426	851	548	7.3
14	0.33	Silica fume, 10	50	450	165	419	837	539	4.7
15	0.39	Silica fume, 10	50	450	195	410	819	527	2.7
16	0.45	Silica fume, 10	50	450	225	403	805	518	0.7

were mixed first followed by the addition of water. Superplasticizer was added at the last stage of mixing. The total mixing time was about 4 min.

(2) *Casting and curing of specimens.* Eight 100 mm cubes were cast from each concrete mixture in which three cubes were for 7-day compressive strength test, three cubes were for 28-day compressive strength test, and the remaining two cubes were for investigating the chloride diffusion after being exposed for 30 days and 60 days in 5 M sodium chloride solution. Further, three 150 mm cubes were cast to perform the initial surface absorption test. Concrete specimens were demoulded 24 h after casting, and then placed immediately in a water curing tank. The temperature of water was maintained at $27 \pm 3^\circ\text{C}$. 28-day standard water curing regime was employed for all concrete specimens before conducting other tests.

(3) *Initial surface absorption.* Initial surface absorption tests were carried out after the concrete was water-cured for 28 days, according to the test method described in BS1881:Part 5. The specimens were oven-dried at 105°C to constant weight prior to the test.

(4) *Chloride diffusion.* On completion of the 28-day water curing, the cubes for the chloride diffusion test were coated on five of the six faces with a coal tar epoxy to simulate unidirectional chloride ingress. The cubes were immersed in a 5 M sodium chloride solution. Following exposure periods of 30 days and 60 days, the specimens were retrieved for laboratory testing. Four holes each with a diameter of 10 mm were dry drilled at the depths of 0–10 mm, 10–20 mm, 20–30 mm, 30–40 mm, and 40–50 mm from the uncoated exposed surface

with a hand drill. The drilling machine was fixed in vertical alignment and was kept normal to the specimen surface. A scale was fixed on the drill to measure the depth of drilling. The drilling machine was started and stopped once it drilled into the specimen by 10 mm. The pulverized materials were then collected before the next drilling. The procedure was repeated until all the samples were collected. The pulverized materials collected from four holes at each depth were combined to give a test sample. The resultant sample was then sieved through a 150 μm sieve. The acid soluble chloride ion contents in each sieved sample was determined in accordance with BS1881:Part 124. This method determines approximately the total chloride content in hardened concrete.

5. Experimental results and discussions

5.1. Series 1 experiments

(1) *Slump and strength development.* The slump, 7-day, 28-day compressive strength of all the concretes in this series are given in Table 4. The slump test results in Table 4 show that at high cement replacement level (30%), in order to maintain the similar slump to the control concrete, higher dosage of superplasticizer was necessary for the concrete with pozzolanic materials. Due to the high water requirement of silica fume, the slump of silica fume concrete was much lower than that of control concrete, except the concrete with silica fume at 5% replacement level.

Table 4

Slump, 3-day and 28-day compressive strength of concrete in series I experiments

Concrete code	Cement replacement material and level	Superplasticizer (l/m^3)	Slump (mm)	Compressive strength (MPa)		7-day/28-day compressive strength ratio
				7-day	28-day	
Control	-	8	198	77.4	96.5	0.80
ZE5	Zeolite, 5%	8	210	90.5	103.8	0.87
ZE10	Zeolite, 10%	8	215	88.1	104.8	0.84
ZE15	Zeolite, 15%	8	220	96.0	110.2	0.87
ZE30	Zeolite, 30%	9.5	200	75.3	101.6	0.74
FA5	PFA, 5%	8	202	88.9	101.5	0.88
FA10	PFA, 10%	8	220	85.1	102.1	0.83
FA15	PFA, 15%	8	215	84.8	106.3	0.80
FA30	PFA, 30%	9.5	210	74.7	103.4	0.72
SF5	Silica fume, 5%	8	225	102	108.3	0.94
SF10	Silica fume, 10%	8	55	101	120.1	0.84
SF15	Silica fume, 15%	8	15	96.8	115.3	0.84
SF30	Silica fume, 30%	13.4	35	95.3	114.7	0.83

Note: Water to total cementitious materials ratio was kept at 0.28.

For both of the zeolite and PFA concretes, the replacement levels from 5% to 15% did not affect the slump as compared with control concrete. It was also found that during the slump test, the concrete with PFA at low replacement levels (5–10%) bled slightly, but no bleeding was observed for the fresh zeolite concrete. It is probably due to its slight increase in viscosity. The test results in Table 4 show that zeolite and PFA concretes had similar slump characteristics.

The compressive strength results in Table 4 show that regardless of the replacement levels, zeolite, PFA, and silica fume increased the 28-day strength. However, at 7 days, when the cement replacement level by zeolite or PFA was up to 30%, the concrete compressive strength was found to have decreased to a level similar to that of the control concrete. This result indicates that when the replacement level reached 30%, both zeolite and PFA concrete developed their early strength at a slower rate similar to that of the control concrete. On the other hand, the strength of silica fume developed much faster irrespective of the replacement level. In terms of the 28-day strength, the replacement level at 15% by zeolite or PFA appeared to be optimum, but for silica fume concrete, the optimum replacement level was at 10%. The 28-day strengths of zeolite and PFA concrete at 15% cement replacement level were, respectively, 14% and 10% higher than that of control concrete.

(2) *Initial surface absorption (ISA) tests.* The results of the initial surface absorption tests are shown in Fig. 1. It can be seen that the replacement of cement by pozzolanic materials can effectively reduce the initial surface absorption. For silica fume concrete, the higher the replacement level, the more notable the reducing effect on initial surface absorption.

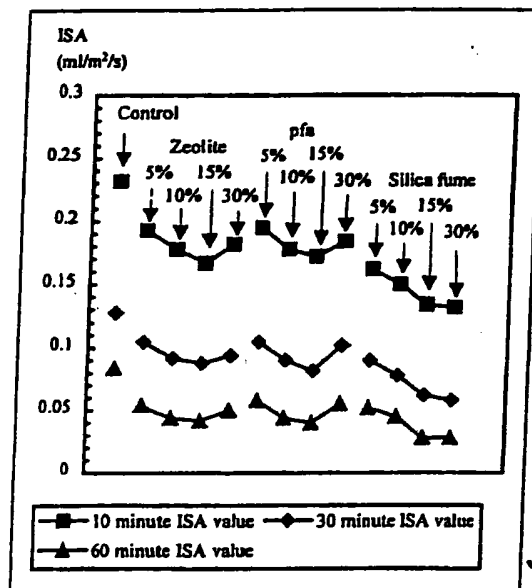


Fig. 1. 10–60 min initial surface absorption of control, zeolite and PFA and silica fume concrete.

But for zeolite and PFA concrete, the most remarkable reducing effect on initial surface absorption occurred at the replacement level of 15%. A similar result was reported by Saricimen et al. for the PFA concrete [7]. In the same figure, it can be found that zeolite had similar effect or slight improvement in the 10-min initial surface absorption when it is compared with PFA concrete. But irrespective of replacement level, the initial surface absorption of silica fume

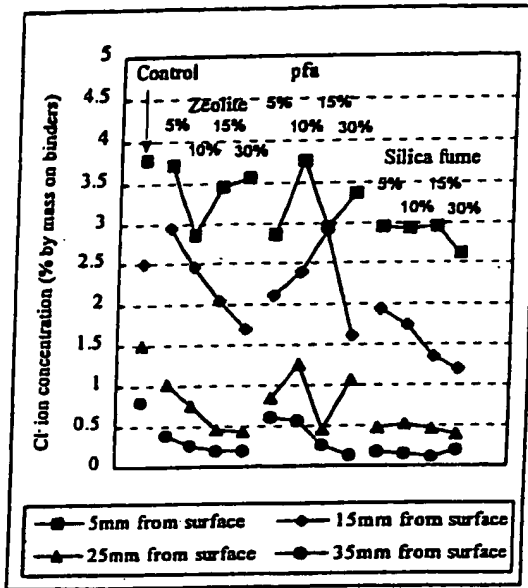


Fig. 2. Chloride contents profile of control zeolite, PFA and silica fume concrete (60-day exposure).

concrete was much lower than those of zeolite and PFA concrete.

(3) *Chloride diffusion*. Chloride content profiles of the concretes at different cement replacement levels are presented in Fig. 2. The figure shows that the chloride concentrations decreased with penetration depth. Generally, the higher the replacement level, the less the chloride concentration at the same depth. The results showed that zeolite has similar effect to PFA on improving the resistance of chloride ingress.

5.2. Test results from series 2 experiments

(1) *Compressive strength and slump*. The test results are given in Table 5. It was found that irrespective of $W/(C + P)$ ratios, the 10% cement replacement by silica fume can increase the 7-day and 28-day strength. However for the concrete incorporating zeolite or PFA, the 7-day and 28-day compressive strength was lower than that of control concrete when the $W/(C + P)$ ratio was increased to 0.45. The slump test results also indicate that the workability of zeolite concrete is slightly superior to that of PFA concrete whereas the silica fume concrete was the least workable.

(2) *Initial surface absorption*. The 10–60 min initial surface absorption of these concretes are given in Figs. 3 and 4. It shows that regardless of $W/(C + P)$ ratios, 10% replacement of cement by silica fume, PFA and zeolite could effect the decrease of the 10–60 min initial surface absorption, with the silica fume concrete having the lowest values. The figure also shows that the initial surface absorption of zeolite concrete is slightly lower than that of PFA concrete.

Fig. 5 shows the comparison of the 10-min initial surface absorption of the concretes at different water to total cementitious ratios. This figure verifies that the higher the $W/(C + P)$ ratio, the higher the initial surface absorption. Regardless of $W/(C + P)$ ratios, the 10 min initial surface absorption of zeolite concrete was similar to or slightly lower than that of PFA concrete but higher than that of silica fume concrete.

(3) *Chloride diffusion*: Chloride content profiles of control concrete, and concrete with pozzolanic materials exposed to 5 M sodium chloride solution for 30 days and 60 days are presented in Figs. 6–9, respectively.

Table 5
7-day, 28-day compressive strength and slump of concretes in series 2 experiment

No.	$W/(C + P)$	Cement replacement material and level	Compressive strength (MPa)		Slump (mm)
			7-day	28-day	
1	0.27	—	91.9	110.0	205
2	0.33	—	72.0	92.9	190
3	0.39	—	50.4	71.3	140
4	0.45	—	40.4	60.3	172
5	0.27	Zeolite, 10%	101.7	115.6	190
6	0.33	Zeolite, 10%	76.4	97.6	180
7	0.39	Zeolite, 10%	53.7	73.1	110
8	0.45	Zeolite, 10%	39.7	57.9	125
9	0.27	PFA, 10%	92.9	114.4	125
10	0.33	PFA, 10%	74.0	94.6	100
11	0.39	PFA, 10%	52.1	70.9	115
12	0.45	PFA, 10%	38.9	56.6	115
13	0.27	Silica fume, 10%	103.8	120.9	25
14	0.33	Silica fume, 10%	91.0	108.6	35
15	0.39	Silica fume, 10%	65.1	83.7	35
16	0.45	Silica fume, 10%	48.7	70.6	60

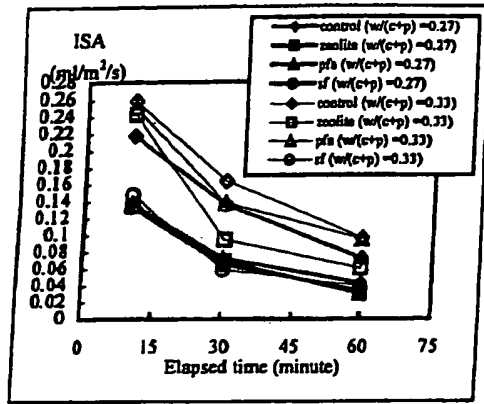


Fig. 3. 10-60 min initial surface absorption of control concrete, concrete with zeolite, PFA and silica fume at $W/(C+P) = 0.27$ and 0.33 .

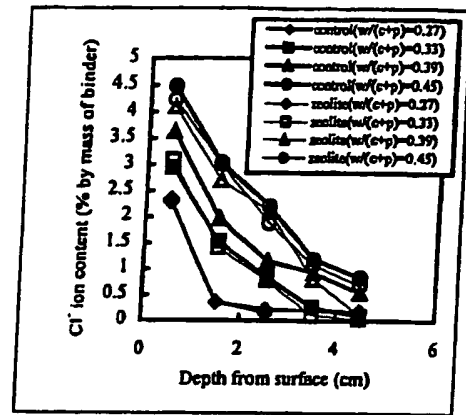


Fig. 6. Chloride contents profile of control and zeolite concrete at different $W/(C+P)$ ratios (30-day exposure).

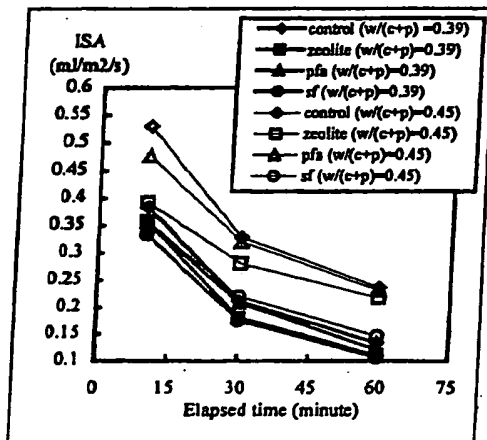


Fig. 4. 10-60 min initial surface absorption of control concrete, concrete with zeolite, PFA and silica fume at $W/(C+P) = 0.39$ and 0.45 .

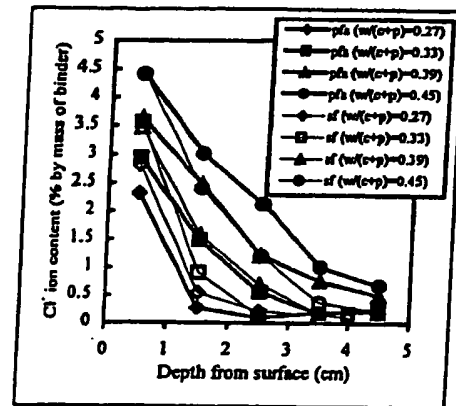


Fig. 7. Chloride contents profile of PFA and silica fume concrete at different $W/(C+P)$ ratios (30-day exposure).

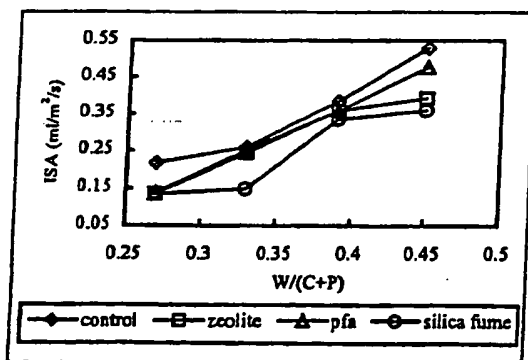


Fig. 5. 10-min initial surface absorption of concrete with zeolite, PFA and silica fume at different $W/(C+P)$ ratios.

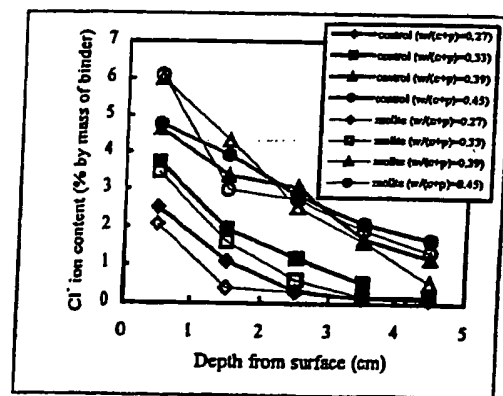


Fig. 8. Chloride contents profile of control and zeolite concrete at different $W/(C+P)$ ratios (60-day exposure).

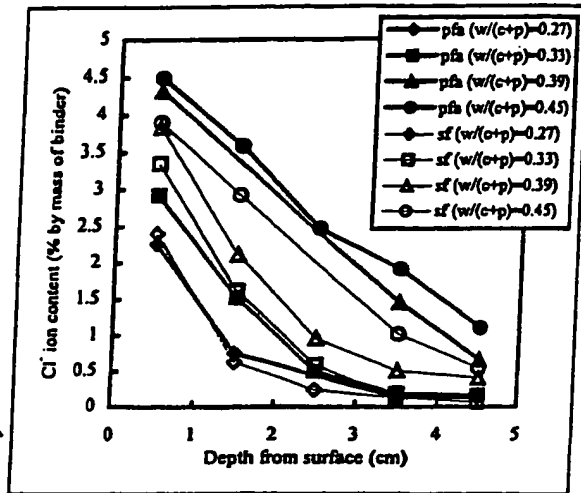


Fig. 9. Chloride contents profile of PFA and silica fume concrete at different $W/(C + P)$ ratios (60-day exposure).

It can be observed that zeolite can improve the resistance of chloride ingress. The effectiveness of incorporating pozzolanic material on improving the resistance of chloride ingress decreased with reducing $W/(C + P)$ ratio.

6. Enhancing effects of zeolite on the microstructure of concrete

The test results from the series 1 and series 2 experiments show that at $W/(C + P)$ ratios from 0.27 to 0.39, partial replacement of cement by zeolite increases the strength of concrete. And irrespective of $W/(C + P)$ ratios, the replacement of cement by zeolite, PFA and silica fume at levels from 5% to 30% can result in the improvement of permeation characteristics of concrete [8]. The lower permeability of pozzolanic material concrete compared to that of ordinary Portland cement concrete can be attributed to the improvement of the microstructure [9].

The comparative study showed that zeolite has a pozzolanic effect on cement concrete. The pore size distribution of neat hardened cement paste (hcp) and hcp with zeolite, PFA and silica fume at $W/(C + P) = 0.30$ was determined by means of mercury intrusion porosimetry and the results are shown in Fig. 10. It can be seen that silica fume hcp has the lowest porosity in the large and fine pore ranges. Although the pore size distribution of zeolite hcp runs parallel to that of PFA hcp, the zeolite hcp still has less porosity than PFA hcp.

It was found that zeolite contained large quantity of reactive or soluble silicon oxide and aluminium oxide:

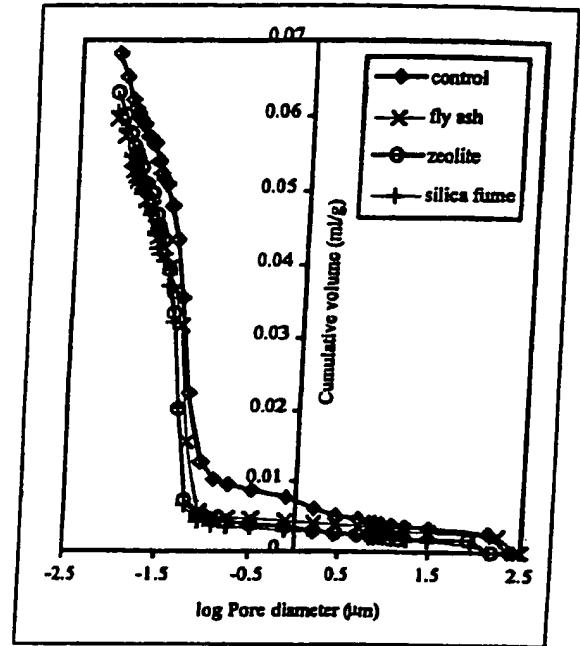


Fig. 10. Pore size distribution of neat hcp, hcp with zeolite, PFA and silica fume (age at 45 days, water to total cementitious materials ratio = 0.30, pozzolanic replacement level = 10%).

$$\begin{aligned} \text{Contents of soluble silica} &= \text{soluble SiO}_2 / \text{total SiO}_2 \\ &= 7.26 / 65.01 = 11.2\% \end{aligned}$$

$$\begin{aligned} \text{Contents of soluble aluminium oxide} \\ &= \text{soluble Al}_2\text{O}_3 / \text{total Al}_2\text{O}_3 = 9.25 / 11.85 = 78\% \end{aligned}$$

The reactive SiO_2 and Al_2O_3 can convert the Ca(OH)_2 liberated during the hydration of cement into C-S-H gel and aluminate, respectively. It was found by X-ray diffraction (XRD) analysis that 10% replacement of cement by zeolite reduced the amount of Ca(OH)_2 in hcp. Table 6 is the XRD test results.

Table 6
 Ca(OH)_2 peak intensity in hcp with and without zeolite (water to solid ratio = 0.25)

Ca(OH)_2 peak position	$2\theta = 18.1^\circ$ (0 0 1 crystal face)		$2\theta = 34.1^\circ$ (1 0 1 crystal face)	
	Age (days)	Intensity (kcps)	Age (days)	Intensity (kcps)
Ca(OH) ₂ peak intensity in neat hcp	3	3.1	3	2.8
	28	3.2	28	2.7
Ca(OH) ₂ peak intensity in hcp with 10% zeolite	3	2.6	3	2.3
	28	2.7	28	2.3

7. Conclusions

1. Zeolite was found to have pozzolanic effect on concrete. The reactive SiO_2 and Al_2O_3 in it converted $\text{Ca}(\text{OH})_2$ into C-S-H gel and aluminates, resulting in the improvement of micro-structure of hardened cement concrete and making the concrete more impervious.

2. Even at low water to total cementitious materials ratio ($W/(C + P) = 0.28$), the replacement of cement by zeolite, PFA and silica fume at levels from 5% to 30% resulted in the increase of compressive strength, and in the improvement of the permeation characteristics of concrete in terms of 10-60 min initial surface absorption and chloride diffusion. It is because the pozzolanic effect further reduced the pore diameters so as to improve strength and permeation characteristics.

3. The replacement of cement by zeolite at 5-15% did not affect the workability of concrete, though it slightly increased the viscosity of fresh concrete.

4. Irrespective of water to total cementitious materials ratios and replacement levels in the two series of experiments, the zeolite was more effective than PFA in increasing the compressive strength, decreasing the initial surface absorption and the chloride diffusion but it was less effective than silica fume.

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